ENSICAEN
ÉCOLE NATIONALE SUPÉRIEURE D'INGÉNIEURS DE CAEN
& CENTRE DE RECHERCHE

ERNEST ORLANDO LAWRENCE
BERKELEY NATIONAL LABORATORY

Lawrence Berkeley National Laboratory
Advanced Light Source – Beamline 1.4

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(Master degree)

ACKNOLEDGEMENT

First of all, I thank:

Dr Michael Martin, who runs beamline 1.4. He had the kindness to accept me in his group and let me work with autonomy on the design of the detector box for the He-3 cryostat. I didn't know much about infrared spectroscopy but he was willing to explain me the basis of this science. He advised me about the different aspects of the box that he wanted for the cryostat's users.

Dr Zhao Hao, associate scientist at beamline 1.4. He helped me when have some problems on the design of the box. He was busy but he never refused to answer my questions.

Dr Gilles Ban, teacher at the EnsiCaen who allows me to carry out my internship at the Berkeley Labs.

Michael Riley, physics teacher of high school in the Comte of Maine, who made a scholar internship in my Laboratory and never refused to help me when I had some problems.

All the scientists, who worked with me during these 3 months of internship.

Besides my project, I really enjoyed my stay at the LBL, appreciated all the people I worked with and spent good moments with them.

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All the drawings are in the same order in the report and the appendices.

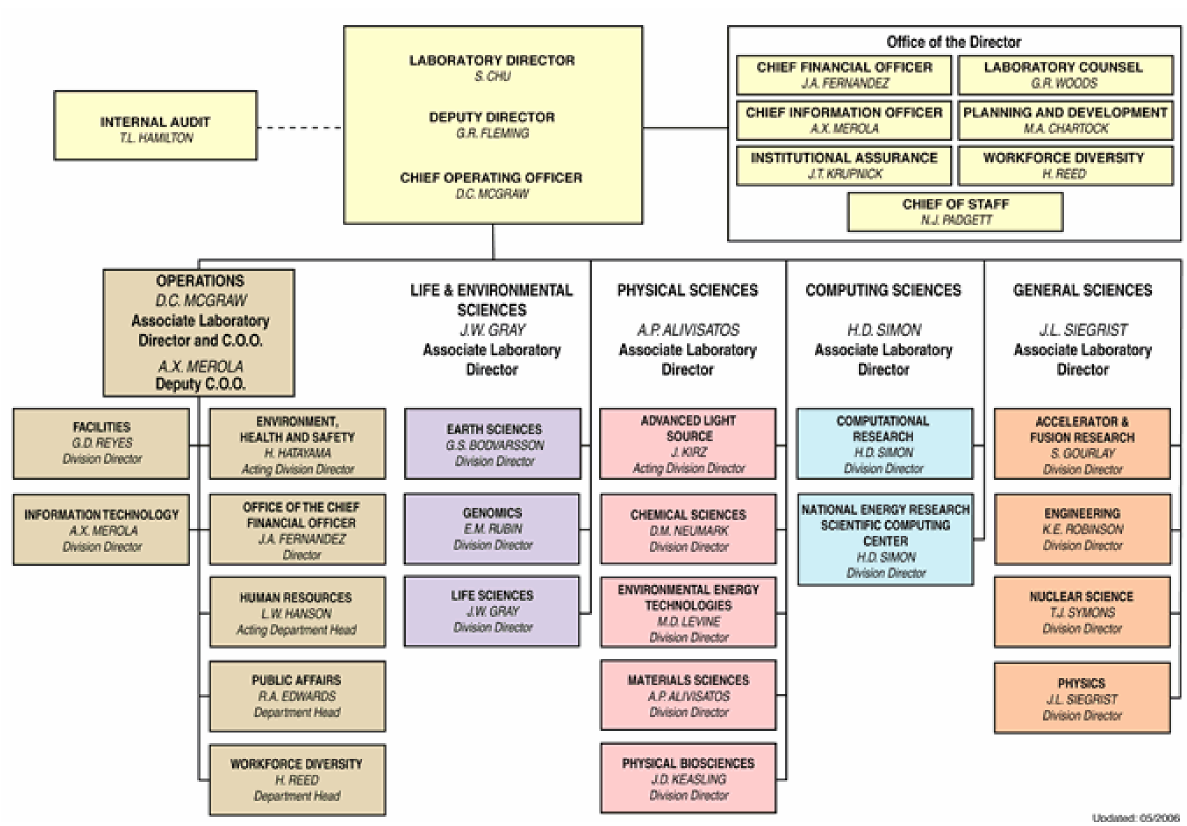
- 3 drawings to explain the principle of the box
- The 12 design drawings of the box
- The 2 design drawings of the two inside plates
- 2 drawings for the parabolic mirror construction
- The drawing of the inside stuff position
- The table for the o-rings
- 3 drawings of different pieces, we have bought

Lawrence Berkeley National Laboratory

Lawrence Berkeley National Laboratory (Berkeley Lab or LBNL) has been a leader in science and engineering research for more than 70 years. Located on a 200 acre site in the hills above the University of California's Berkeley campus, adjacent to the San Francisco Bay, Berkeley Lab holds the distinction of being the oldest of the U.S. Department of Energy's National Laboratories. The Lab is managed by the University of California, operating with an annual budget of more than $520 million and a staff of about 3,800 employees, including more than 500 students.

Berkeley Lab conducts unclassified research across a wide range of scientific disciplines with key efforts in fundamental studies of the universe; quantitative biology; nanoscience; new energy systems and environmental solutions; and the use of integrated computing as a tool for discovery. It is organized into 17 scientific divisions and hosts four DOE national user facilities.

The Lab was founded in 1931 by Ernest Orlando Lawrence, winner of the 1939 Nobel Prize in physics for his invention of the cyclotron, a circular particle accelerator that opened the door to high-energy physics. It was Lawrence's belief that scientific research is best done through teams of individuals with different fields of expertise, working together. His teamwork concept is a Berkeley Lab legacy that has yielded rich dividends in basic knowledge and applied technology, and a profusion of awards. Today there are ten Nobel Laureates associated with Berkeley Lab.

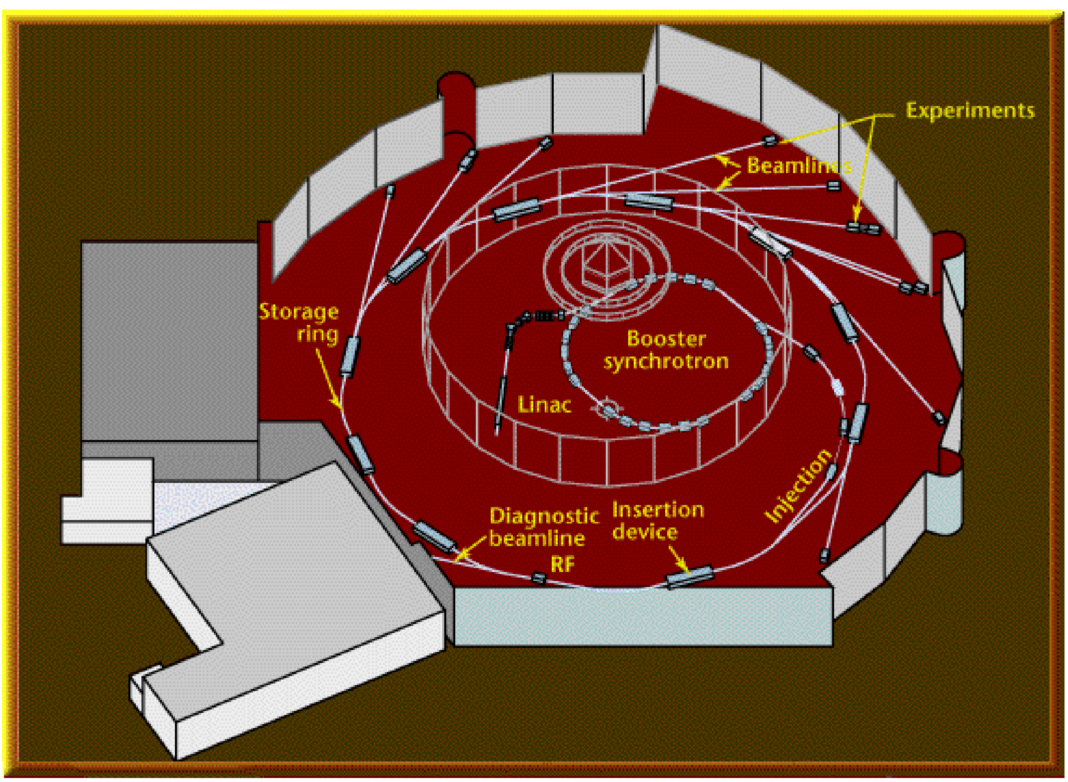


The ALS

The Advanced Light Source (ALS) is one of the 4 national research facilities at LBNL. The total ALS staff is 185 people which welcome more than 1900 visitors and researchers per year.

The main part of the synchrotron is the electron storage ring. It's a vacuum chamber which carries an electron beam traveling through it at nearly the speed of light and at a nominal energy of 1.9 GeV. The storage ring is roughly circular with 12 arc-shaped sections joined by 12 straight sections. As the electron beam curves in a magnetic bending section of the ring, it emits light collected by beamlines which deliver the light to the experiment stations. Many straight sections have undulators which are sources of narrow bandwidth high-brightness soft x-rays.

One advantage of this bright source is that many scientists can use the synchrotron at the same time. The lifetime of the beam in the ring is long; after 8 hours approximately half of the beam current is lost so the ring is refilled. The electron beam is physically small which means that the light emitted can be refocused to a very small area. The ALS produces light in the x-ray region of the electromagnetic spectrum that is one billion times brighter than the sun. This extraordinary tool offers unprecedented opportunities for state-of-the art research in materials science, biology, chemistry, physics, and the environmental sciences. Ongoing research topics include the electronic structure of matter, protein crystallography, Ozone photochemistry, x-ray microscopy of biological samples, lithography, and optics testing.



The Beamline 1.4

The beamline acquired 2 spectrometers with microscopes for the infrared Spectro-microscopy. Biologists and researchers come from around the country to perform various experiments using the equipment at the beamline.

Scientists use light of different frequencies to study materials. Many modern experiments require the use of x-rays, but the beamline 1.4 uses light in the infrared region for experiments in environmental, biological, and material sciences. This is because infrared light interacts with chemical bonds and subatomic particles differently than other wavelengths of light.

Scientists study samples using transmission or reflection properties to obtain a spectrum of their sample. The synchrotron IR beam is focused to a small spot (< 10 microns) which allows the study of small regions in a sample.

The beamline has another spectrometer for very low temperature experiments (4 Kelvin). This spectrometer is useful for investigating the behavior of semi-conductor.

The unit of measure for infrared spectroscopy is wave numbers, versus wavelength. Wave number is the inverse of wavelength in centimeters. So, wave number is proportional to the energy of the photons.

Introduction

The beamline1.4 has acquired a cryostat designed by the firm Janis in the year 2004. It is supposed to reach a very low temperature, around 0.3 K. Some samples need to be cooled to such temperatures so that they are worth studying. That's the case for semi-conductors with a very small band gap. To study it when it behaves as an insulator, it must be cooled- room temperature generates enough energy so that electrons jump across the band gap allowing conductivity. Some other materials such as superconductors should be studied at low temperature also, since superconducting transition temperatures can be very low.

The cryostat has two optical accesses and different windows can be used. Each window will transmit different wavelengths of the light. Scientists can carry out transmission or reflection measurements. Obviously, the sample will be affected by room temperature radiation, especially when a transmission measurement is in place since there will be windows on two sides of the sample.

Despite my limited expertise in the field of infrared spectroscopy, I do have extensive knowledge in the areas of gamma and X-ray spectroscopy. During my assignment at the ALS, I learned the basics of infrared spectroscopy and the use of the software OMNIC, but I mainly work on the design of the adapted box for the cryostat.

However, I experimented on basic substances for showing me the application of the software and the different methods of the spectroscopy such as Attenuated Total Reflection (ATR)

Last year, Yangsanak Bounthavy, second-year student of Ensicaen, did great work on the assembly and the test of the cryostat. After this, the beamline had to continue the cryostat project for real experimentations on semi-conductors. My work began with the task of designing, assembling and setting up the box connecting the cryostat to the detector. Measurements were taken on the cryostat for deciding dimensions for the box. After this, drawings were made for the Berkeley Labs and ALS workshops. Then I had to decide, with the support of Michael Martin, what must be ordered for the construction of the box.

For the design of the box, many drawings could have been created by a company employed to make them, in such the same way a basement is designed. However, the price quoted by the firm was too expensive for the beamline 1.4 budget and the dimension of that box is not the same as our cryostat. That's why Michael Martin told me that this subject is a good project for a three months internship.

Information: For more precision on all the different little pictures I have included in the report, you can have a look on the Appendices where all the drawings are in the original size.

1. <u>The He-3 cryostat:</u>

1.1. Some physics principles

Consider a liquid in a beaker. It is always evaporating to generate vapor which will be in equilibrium with this liquid. And the vapor is always condensing to be in equilibrium with the liquid. At equilibrium, the rate of evaporation and condensation are equals. So if we remove some vapor, the liquid will evaporate more to re-established the equilibrium. The liquid needs energy in order to evaporate. The energy is taken from the beaker. As a result, the beaker will be cooled down.

Heat can be transferred by 3 different ways: conduction, convection, and radiation. To keep a cryostat cool, we want to avoid those heat transfers. In a vacuum, conduction and convection can't take place. To eliminate radiation from room temperature, we can use good reflection materials or shield the cryostat from room temperature with various heat shields which are at colder temperatures.

- Nitrogen gas condenses at 77 Kelvin and freezes at 63.
- Liquid He-4 condenses at 4.2 Kelvin. Helium does not freeze at atmospheric pressure.
- Helium-3 condenses at 3.2 Kelvin.

1.2. The Janis He-3 cryostat

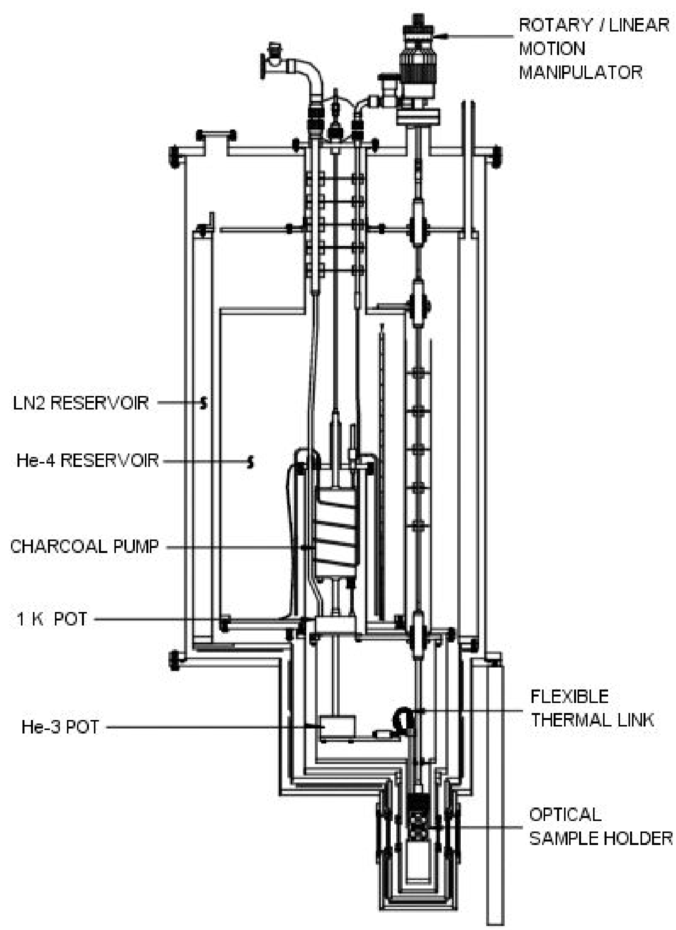
The cryostat consists of three reservoirs inserted one in each other:

The outer reservoir sealed the cryostat and is under vacuum. This reservoir is at room temperature and consequently emits radiation at 300 K.

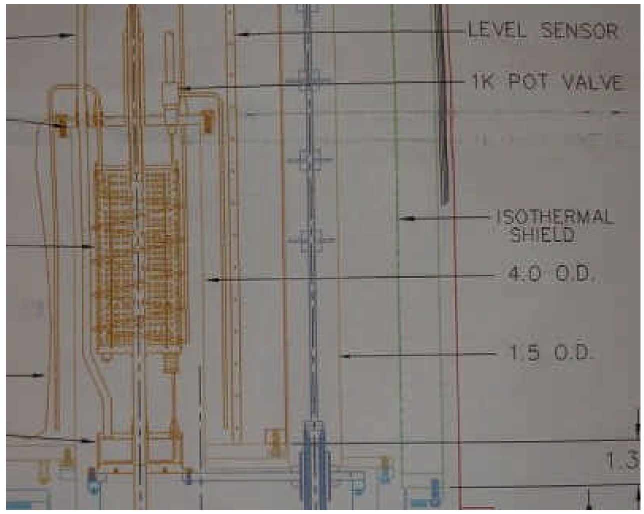
Inside, there is nitrogen reservoir. It is at 77 K and shields the inner reservoir from the room temperature radiation.

The Helium reservoir is at 4.2 K and shields the sample and He-3 systems from the 77-Kelvin-Nitrogen reservoir radiation.

The cryostat also consists of a He-3 insert which includes the charcoal sorption pump, the 1 K pot and the He-3 pot.



The 1 K pot can contain liquid He-4 and is used to condense He-3 gas into the He-3 pot. He-4 is introduced into the 1K pot through the 1 K pot needle valve (1 K pot valve on figure 2). The flow rate of He-4 can be adjusted from the top of the cryostat. When the needle valve is opened, some liquid Helium will be introduced into the 1 K pot. We then pump on the 1 K pot to remove the He-4 vapor, thus causing the liquid helium to evaporate and re-establish some lower vapor pressure.



The vapor then reaches equilibrium with the liquid again. While evaporating, the 1 K pot will cool down ultimately to approximately 1 Kelvin.

He-3 is condensed into the He-3 pot which is installed below the 1 K pot. The charcoal pump absorbs the He-3 gas from the He-3 gas reservoir in a first step. Then, by heating the charcoal, He-3 gas is desorbed towards the bottom. When the gas is in contact with the 1 K pot which has been cooled down as described above, it will condense and be collected in the He-3 pot. So, there will be liquid He-3 at around 3 K.

To reach a lower temperature, the charcoal pump is cooled down again. When it is cooled, it functions as a vacuum pump on the He-3 pot and will reduce the vapor pressure of He-3 liquid. So liquid He-3 will evaporate to re-establish the liquid-vapor equilibrium and the He-3 pot will be cooled down. A cooper thermal strap links the He-3 pot and the sample holder. Thus the sample holder reaches also a low temperature.

There are 4 sensors in the cryostat to measure the temperature at specific locations:

- TA is a diode sensor that measures the charcoal temperature.
- TB is a diode sensor reads the 1 K pot temperature.
- TC is a Ruthenium Oxide resistor which gives the temperature of He-3 pot in K Ohm.
- TD is a Ruthenium Oxide resistor that measures the temperature of the sample holder in Kohm.

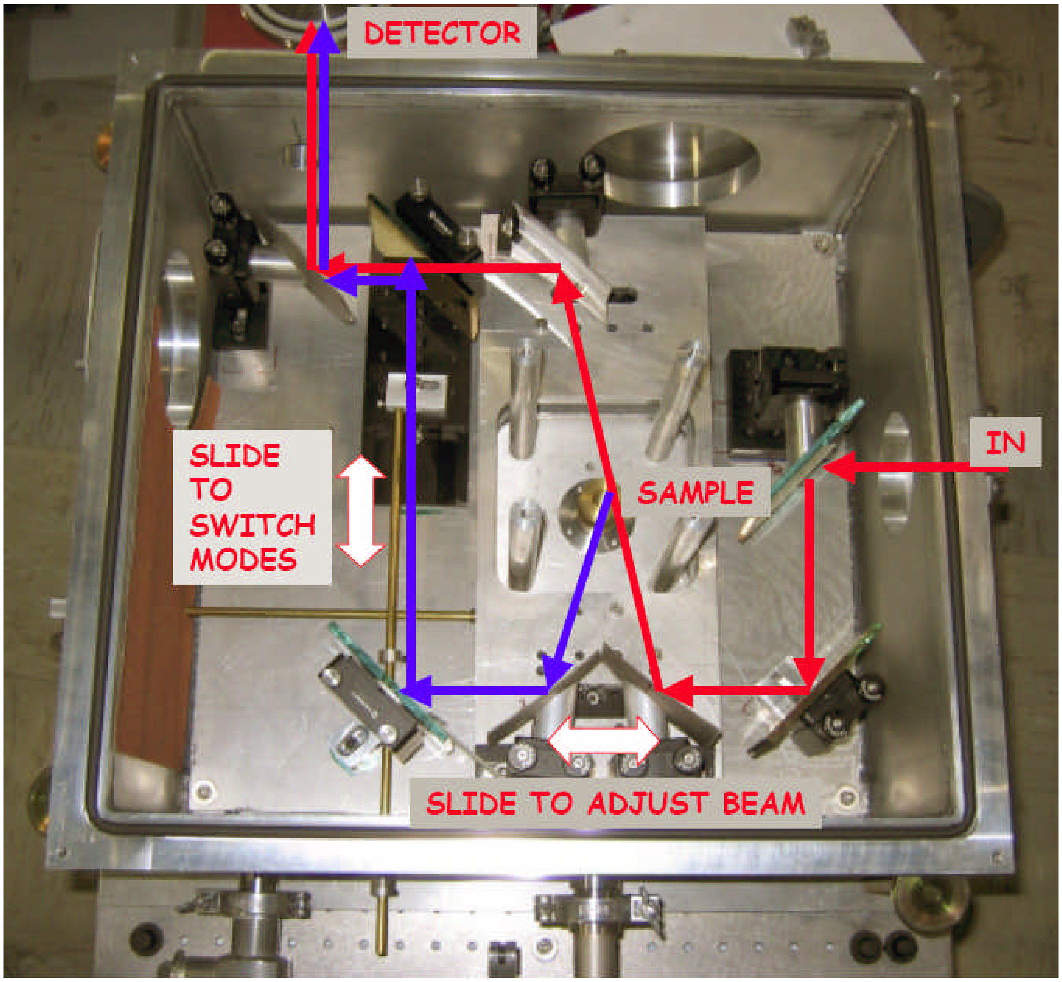
2. <u>The schedule of conditions</u>

2.1. The principle of the box

The entrance to the box was designed on one box side, allowing a path for the beam to reach the sample for future experimentations (transmission or reflection). After, the beam changes direction with a flat mirror directed at a ninety degrees angle. It then deviates by a second flat mirror with a ninety degrees angle.

Then, the beam is focused on the sample by an off-axis parabolic mirror. Here, the sample receives the majority of the source beam and diffuses the light by transmission and reflection.

In the case of reflection or transmission, it's the same process: the light is focused by a second off-axis parabolic mirror with the same focal distance as the first. Then the beam is directed by one (two) flat mirror in the case of transmission (reflection) to a third off-axis parabolic mirror with a different focal distance adjusted to the distance between the parabolic mirror and the measurement point of the final detector (a bolometer).



2.2. *What do we need for the box?*

After the explanation of the box principle, we can define the stuff that we need for the box:

- The box with a cover adapted to our cryostat.
- 1 entrance and 1 exit to make the vacuum inside the box.
- 1 entrance and 1 exit for the beam.
- 1 plate as a basement for all the optic stuff.
- 1 smaller plate for the slide to adjust the beam.
- 2 Plexiglas windows on two opposite sides of the box to adjust all the optic stuff without putting off the box from the cryostat.
- 8 optic supports for the 8 mirrors with an adjustment for the height and the direction of the mirror itself.
- 4 flat mirrors.
- 3 off-axis parabolic mirrors for the beam focus on the sample.
- 1 off-axis parabolic mirror with a longer focal distance for the focus in the detector.

However, we must choose the different box optic stuff with caution to maximize the light power of the beam:

- The plate, where the three off-axis parabolic mirrors are located in the center of the upper picture, must move a little (approximately 1 inch) to adjust the focus of the beam on the sample. That's why we need 2 slides on the both side of the plate and an exit to move the plate without opening the box.
- We need also another slide for the last flat mirror to switch modes (transmission or reflection) and its exit.
- All the different mirror supports of our box must move too to adjust the directions of the beam.

We also need 3 O-rings for doing the vacuum in the box:

- 2 between the 2 Plexiglas windows and the box wall
- 1 between the cryostat and the box wall

Future users of the cryostat want an entrance to excite the sample, which will be located between the two off-axis parabolic mirrors on the picture. Finally, an entrance for the light is needed.

After making an inventory of all the equipment needed, the biggest part of my work ensued: making all the drawings and giving the workshop plans to manufacture the different pieces of the box. For all the pieces and drawings of the box, I discussed the different ways to manufacture and plan the box with Michael Martin.

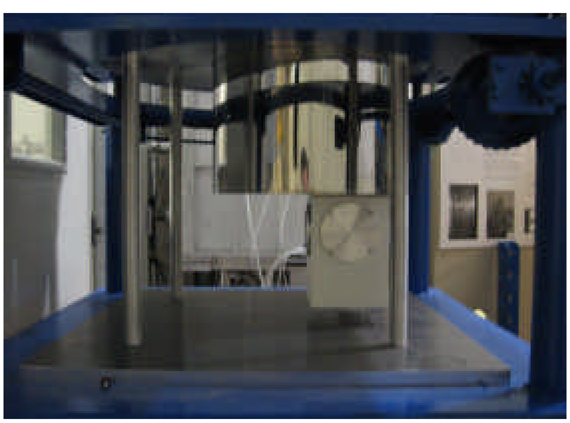
3. <u>The design of the box</u>

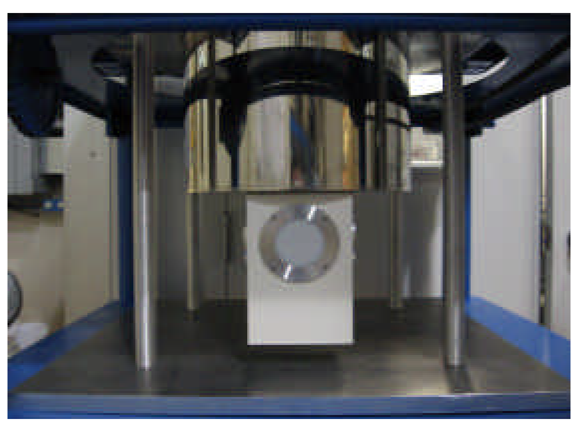
3.1. The dimensions

First, I took all the measurements to dimension the outside box connecting the detector to the cryostat. For that, I have taken the size between the four blue bars of the cryostat support:

- Width 25 inches
- Length 25 inches
- Height 15 inches

As you can see on the above picture, the cryostat is posed on a removable support. The four grey aluminium bars can be removed with the aluminium plate under them. The white cube with the windows, at the end of the cryostat, has 5 inches for the length, 5 inches for the width and 7.5 inches for the height. There are 4 outside issues on this white cube. We'll use two of them for the beam. The cryostat roll above the cube has a diameter of 12.5 inches.



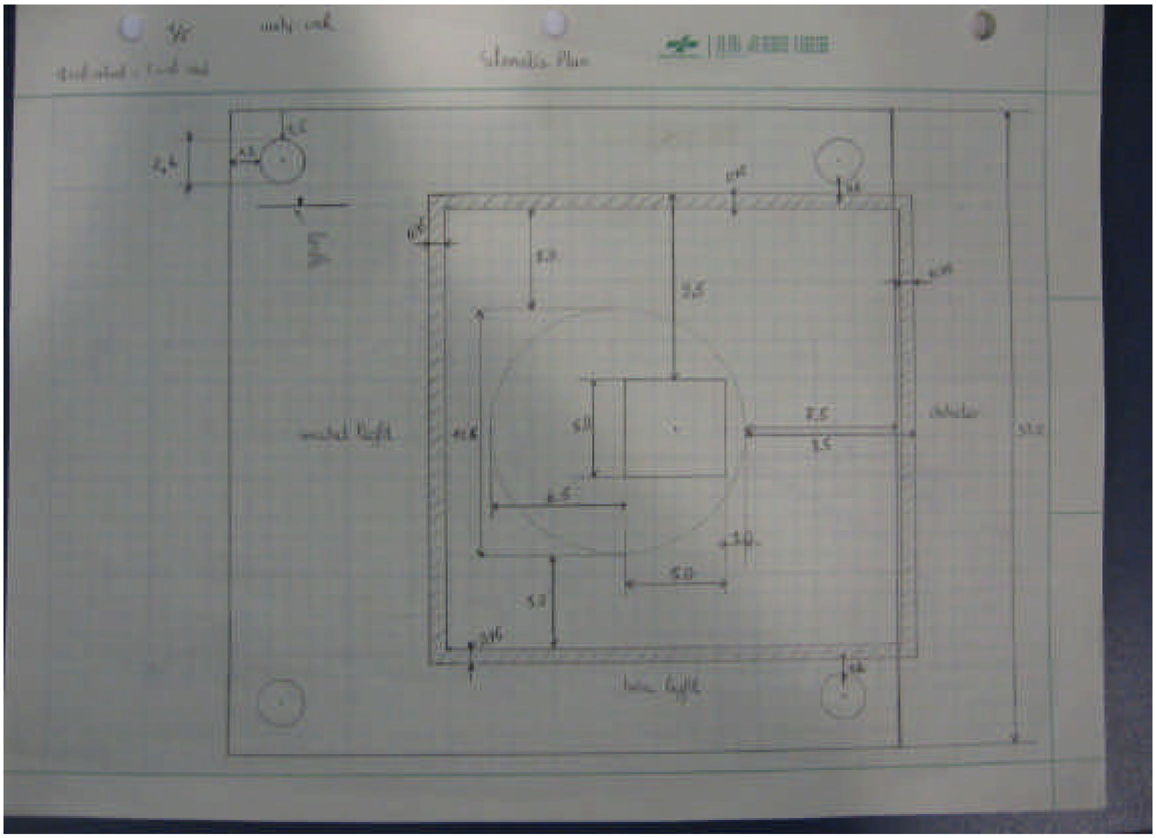


In consequence, I must adapt the box dimensions with these measurements.

I took these distances for a best utilisation and manipulation of the cryostat:

- Width 24 inches
- Length 24 inches
- Height 12 inches

The below picture represents the position of the box under the cryostat, assuming the calculations are correct.

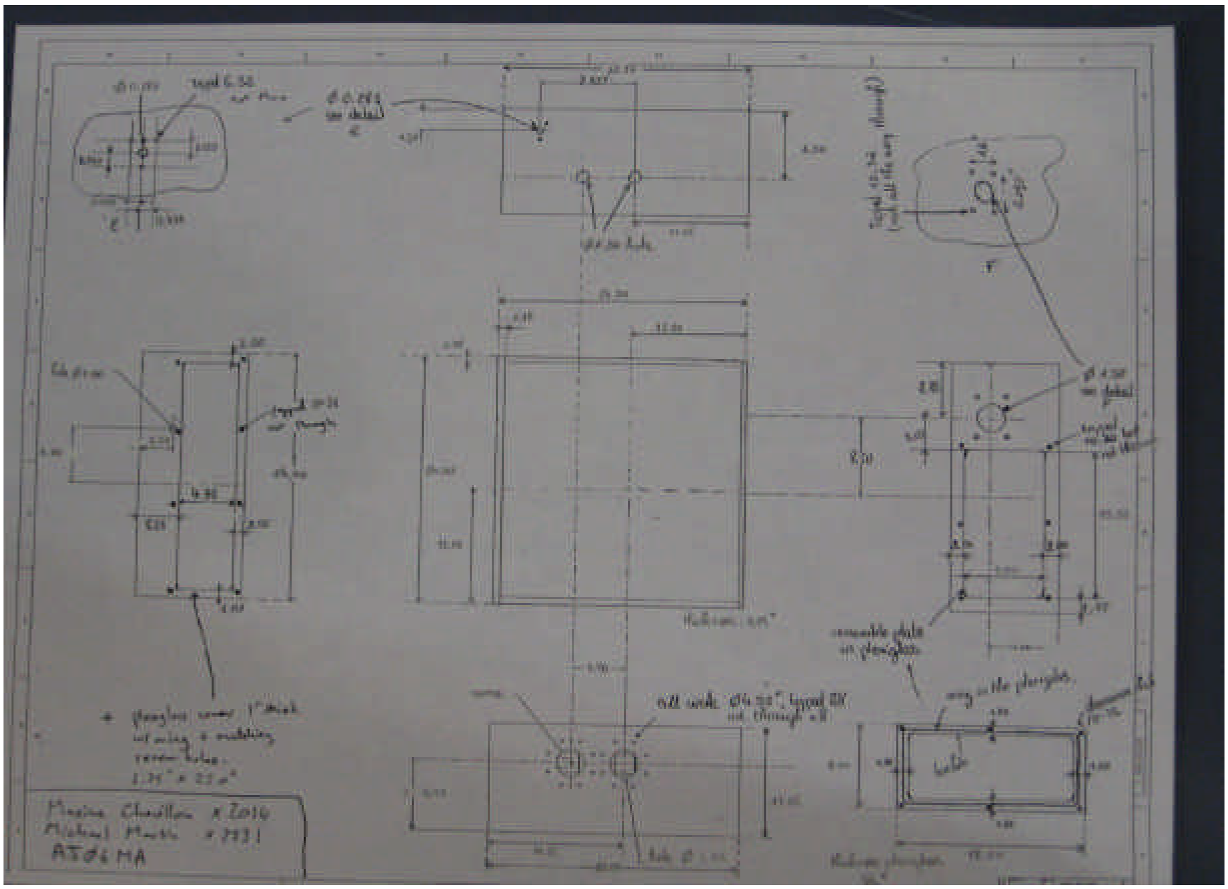


During calculation, it was discovered that the handle of the cryostat support required designing. The box was put into this position because the handle is 11 inches upper from the basement support where the box must be positioned.

We must also take care of the position of the different windows, entrances and exits because the blue bars of the cryostat support can block them.

3.2. The explanation of the box drawings

After choosing what was needed for the box, the drawings were taken to the firm to draw an outline of the plan. I attempted to customize the firm's drawings for our use and our box dimensions. However, plans don't match well for the workshop, as shown below. That's why some other precise drawings of each side of the box must be made.



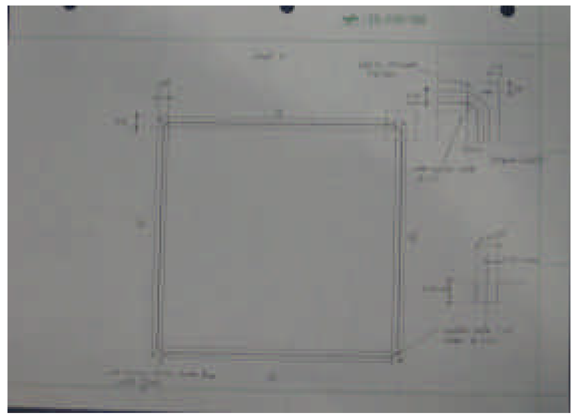
A drawing of the other side allowed me to define and place all the materials required, in their approximate location on the box. The precise drawings were then made. All of the individual choices made with Michael Martin will not be covered in this paper because there are too many extraneous details.

The most important design considerations for the detector box are presented below:

- All the plates on the following drawings have a thickness of 0.75 inch.

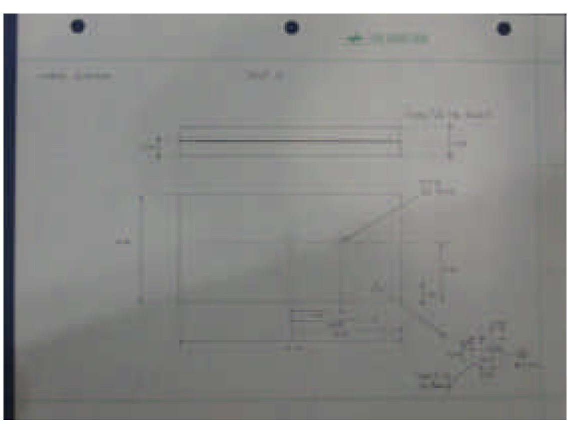
- The bottom plate does 24 inches on length and width. There are 4 screw holes (tapped but not through) in each corner of the plate for the basement plate of the optic stuff.

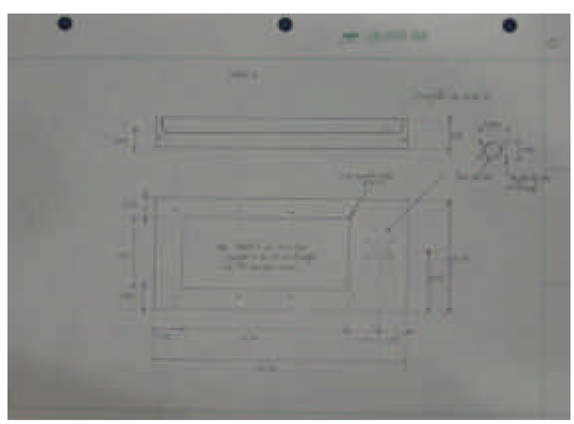
- This picture is the drawing of the box with all of the plates welded together. At the end, an o-ring hole must be designed in the top of these plates for the cover plate. To define the depth and the width of an o-ring hole, I used a conversion table, listed in the Annexes. There are also four screw holes to fix the cover to the box and press the o-ring.



- In the first side plate, there are two 1.00 inch diameter holes for making the vacuum in the box. In consequence, we need two connectors for these holes: Base plate to Kwik-Flange KF of the MDC-vacuum firm. There is also a 0.281 inch diameter hole for the slide of the off-axis parabolic mirrors plate. For this hole, we have chosen a rotary feed through that the beamline 1.4 has in its cupboards.

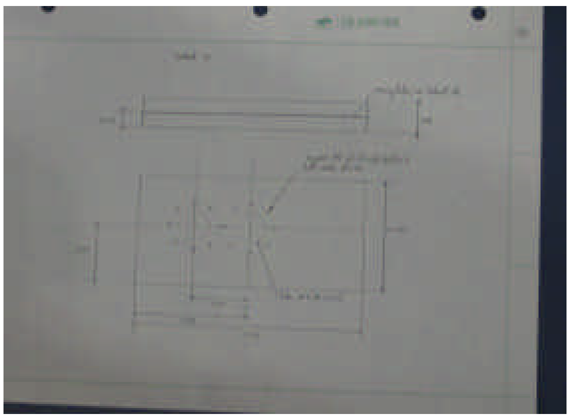
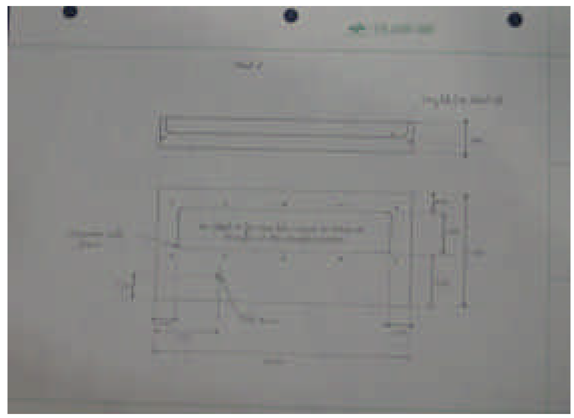
- The second side plate is for the exit of the beam to the detector after its interaction with the sample at 0.3 Kelvin. For this exit, there is a 1.50 inch diameter hole for a specific bolometer connector that the beamline 1.4 has. This side plate has also a big through hole which does 7.00 inches on height and 15.50 inches on length for the littlest Plexiglas window. Around this hole, there are 8 screw holes to fix the window on the aluminium side plate.



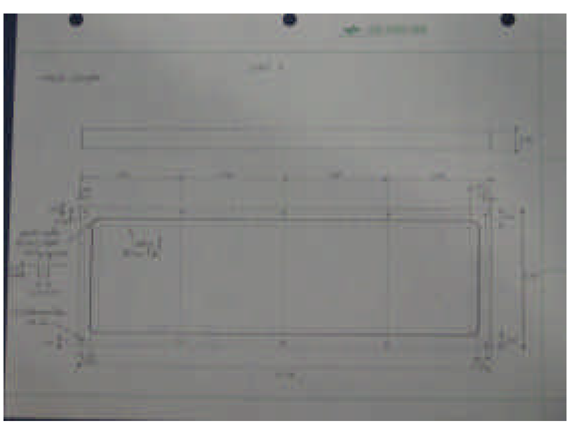
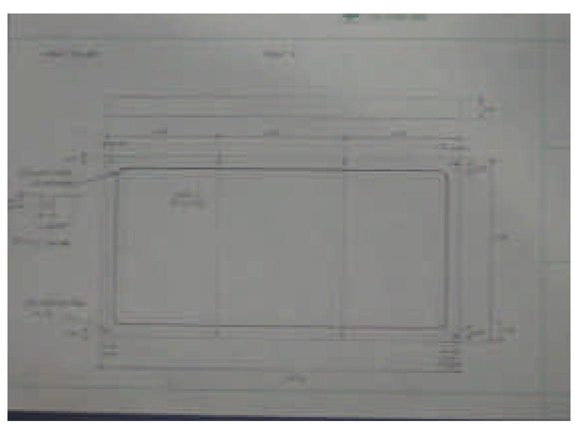


- The third side plate is for the entrance of the beam. However, we decided to add another entrance hole for an issue in the case of problems with the first beam entrance or the setup of the beam on the first off-axis parabolic mirror. These two 3.26 inches diameter holes have an 8-bolt circle (tapped but not through) for the Claw Clamp connector.

- In the fourth and last side plate, there is a 1.00 inch diameter hole for the push-pull slide of the last flat mirror plate. This side plate has also a big through hole of dimensions 4.25 inches high and 20.00 inches long for the biggest Plexiglas window. Around this hole, there are 10 screw holes to fix the window on the aluminium side plate.

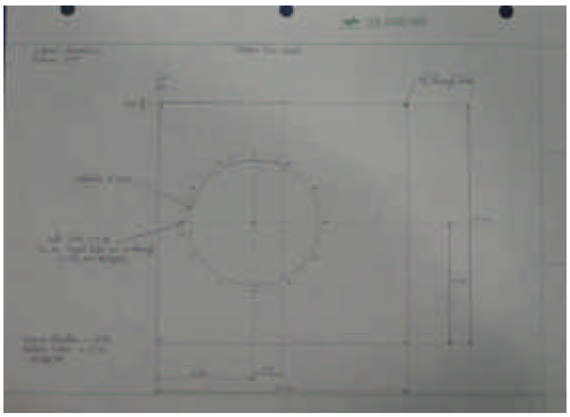



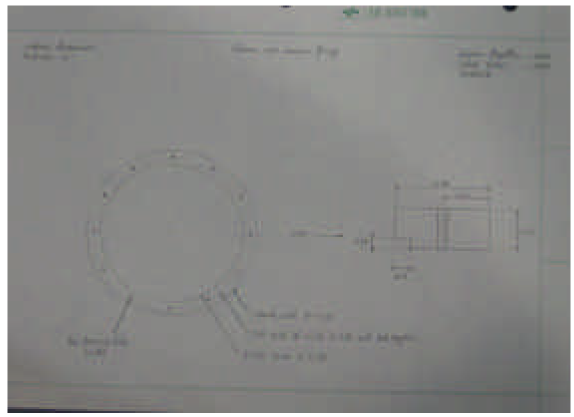
- The two Plexiglas windows were included to improve the process and make for a quicker change of the inside optic stuff setup. The first window has dimensions of 17.50 inches long, 9.00 inches wide and 1.00 inch thick. The second window has dimensions of 22.00 inches long, 6.25 inches wide and 1.00 inch thick. They have the same structure for the o-ring and the screw hole. There are 10 screw holes in the first window (8 for the second) for press the o-ring and make a good isolation for the vacuum. The o-ring groove is positioned at 0.625 inch from the Plexiglas edge. We calculated the o-ring size with the most precision possible given the measuring tools available. However, McMaster-Carr carries some round o-rings of ¼ inch thickness, even though his is ½ inch longer. We then ordered a 14.5 inches diameter o-ring and 15.5 inches diameter o-ring.

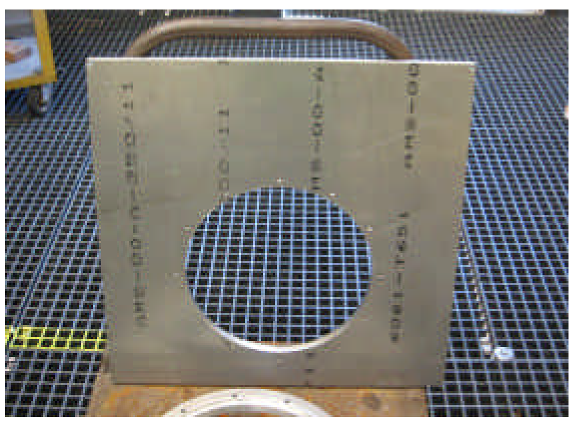
- The last plate is secured onto the cover plate well. There are four clearance holes to close the box. The 12.6 inches diameter hole is for put the useful part of the cryostat in the box. This hole is surrounded by a 13.75 inches diameter bolt circle: screw holes each 30 degrees (tapped holes but not through). We have also ordered a 12.5 inches diameter round o-ring of ¼ inch thickness.

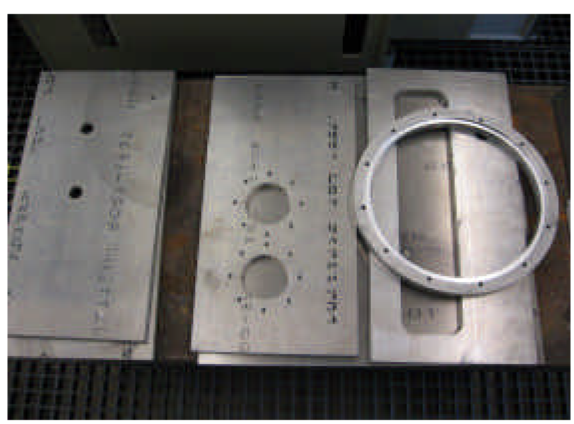
- The circle plate is used for sealing the cover o-ring. There are as well some screw holes each 30 degrees to press it on the o-ring. To define the depth and the width of an o-ring hole, I used a conversion table which is in the Annexes.





Finally, the main Berkeley Labs Workshop has not finished the entire box when I left Berkeley. But they have the different sides of the box which will be cleaned and welded together after my leave:



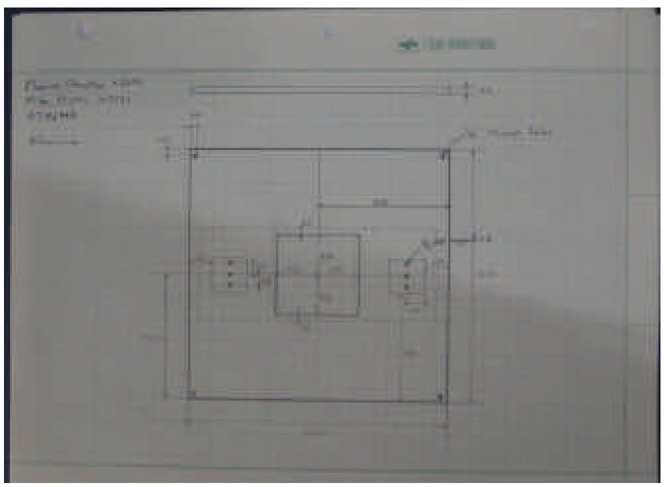


4. <u>The inside stuff</u>

4.1. The plates

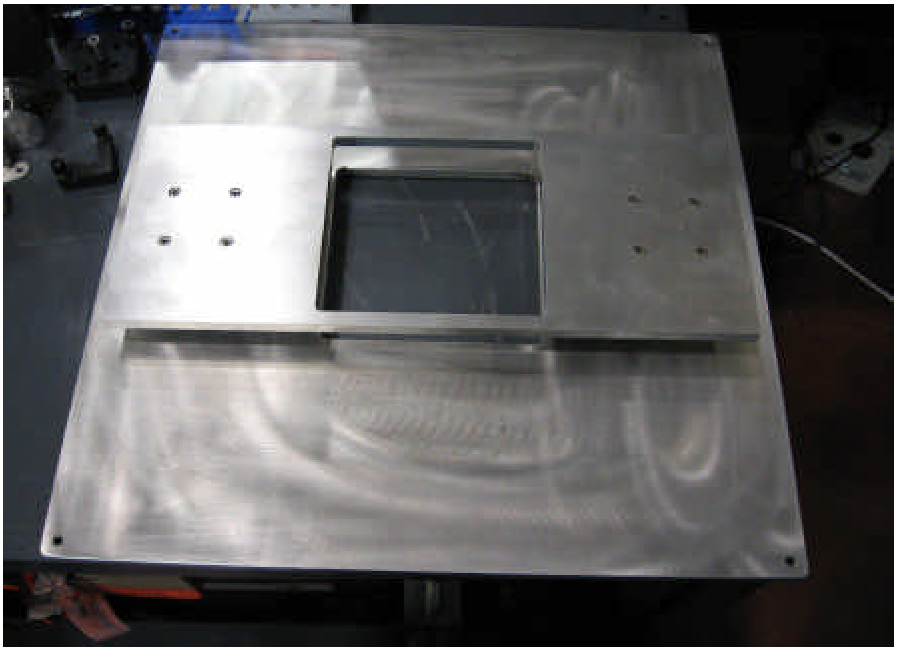
The first step to create the inside optic environment is to design and send the plans of the two inside plates to the workshop of the Advanced Light Source.

The first plate we described is the inside basement plate, ½ inch thick. It is ½ inch above the background box plate. We will intercalate four small supports between this plate and the background of the box on each corner inside to fix it. This plate is useful to test the position of the different optic pieces we will fix on. The two inside plates will slide together to position precisely the beam on the sample. For that, we ordered 2 slides from the firm Newport optics and put the position of the screw hole on the manufacturing plan. The 7 by 7 inches hole in the center of this plate was created for the place of the cryostat windows and the sample in it.



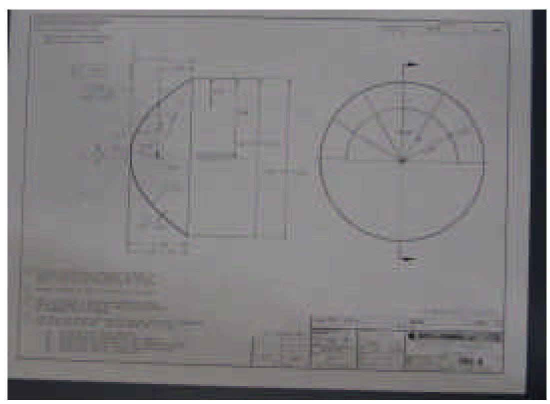
This second plate is the upper plate which will be moved with the 2 slides. There are also the center hole for the cryostat windows and 8 screw holes to fix the slides on it. Its thickness is ½ inch.

Finally, we received these results from the ALS workshop:



4.2. The choice and position of the optical components

Now we can define precisely all the components needed to direct and focus the beam on the sample and then exit it on the bolometer. All the following materials were chosen with budgetary considerations.



First, we have chosen the mirrors:

- First, we ordered the 8 flat mirrors from Edmund Optics.

- Second, we found some off-axis parabolic mirror with the adapted focal distance. Then we needed to find another way to obtain these off-axis parabolic mirrors. For that, we followed the advice of Michael Martin's colleague: we bought a large parabolic mirror from Opti-Forms and cut this mirror to have the focal distance we needed.

The next step was to define the support and the mount for the different mirrors- we bought the following from Thorlabs:

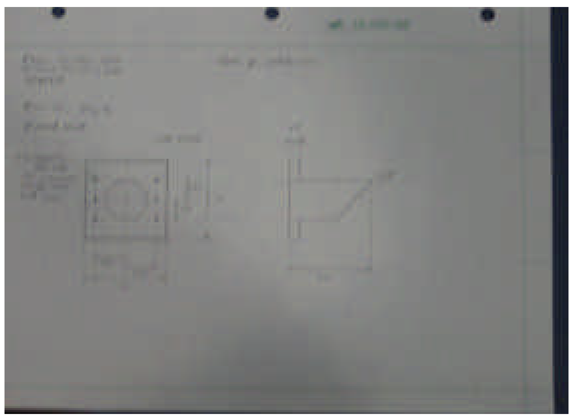
- Kinematic Platform Mount (2 by 2 inches).

- Post Holder with Spring-Loaded Thumbscrew of 2 inches.

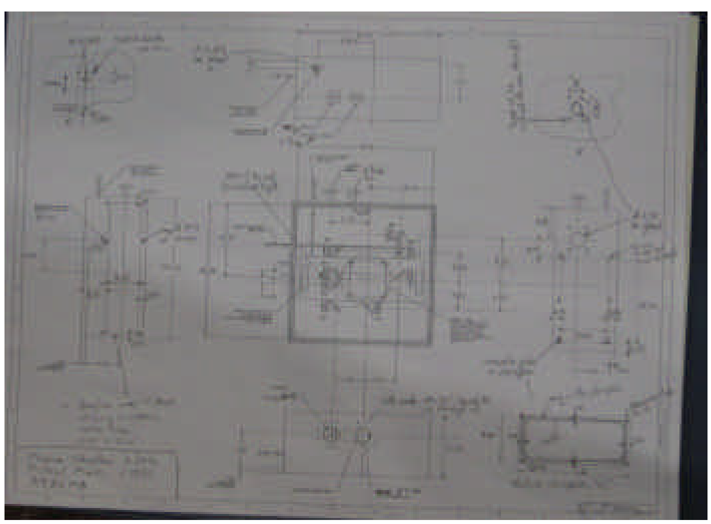
- Post Holder with Spring-Loaded Thumbscrew of 3 inches.

For the flat mirrors, it’s easy to do a 90 degree angle with the mount. But for the 3 off-axis parabolic mirrors on the center plate, we must create a support for these mirrors because their utilization needs to be with a 50 degree angle approximately.

For these supports, I made the next drawing:



Finally, I have positioned approximately the position of each mirror on the box plan drawing to prepare the different stuff, we have bought for our box.



Conclusion

My project during this internship was to design the drawings to manufacture the box connecting the cryostat and the detector by the Berkeley labs workshop. I started by defining what we needed to accomplish that. Then we designed all the drawings and ordered all the components we wanted for our box. We finished by choosing and buying the inside optical equipment. However, the project isn't finished and someone must continue it to arrive at a useful application for the beamline.

Concerning this project, it was interesting to discover something else that I was taught in my engineering school such as electronics or spectroscopy in x-ray region. The work in itself was also interesting in different point of views. Designing the box drawings allowed me to learn some new design techniques.

Moreover, working on a concrete problem made me learn several things. Even though that was something new for me, I could use tools I knew to find solutions like cutting a big parabolic mirror to make several little ones with the focal distance we need. Now, I understand the principle of a cryostat thanks to what I learnt 2 or 3 years ago in thermodynamic and the process for manufacturing equipment such as this. I was already aware of much of this, but here I could see for real that besides the tools we learn in our studies, what is really useful is the scientific approach we acquired to solve problems. Then, talking with people and exchanging ideas, I came up to the view factor.

This internship was a great experience. It was a real pleasure to work in this ALS at the beamline 1.4. I could work with nice people in a good atmosphere. Carrying out this internship in a foreign country allows me to be immersed in a different culture and faced the problem of living and working entirely in a non-native language- English. I met several interesting people in and out of the lab such as researchers, students in physics, but also people not connected with science.

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INTERNSHIP APPENDICES

May 1 – July 28, 2006

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